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EFFECT OF THE PHASE COMPOSITION ON THE DURABILITY OF CERAMIC FACING OF THE SHAKHI-ZINDA ENSEMBLE IN SAMARKAND

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X-ray phase and electron-microscopic methods showed that wollastonite and anortite determine the strength of ceramic facings of the Shakhi-Zinda ensemble. In addition, the isometric pores and oval closed porosity present in the ceramic facing of the Shakhi-Zinda ensemble increase durability.

Key words: Shakhi-Zinda mausoleum, ceramic facing, phase composition, durability, x-ray diffraction pattern, electron-microscopic method, wollastonite, anortite, slit-shaped pores, monticellite, porosity.

The problems of the durability of structures and buildings and lowering the cost of capital repairs are very topical and are due to the scales of commercial, residential, and individual construction.

In the production of ceramic materials, special importance is given to phase compositions and porosity, since these are the main factors affecting the operational properties of articles [1 – 3].

The task of the present work is to determine by x-ray phase and electron-microscopic analysis the phase composition and porosity in the ceramic facings of the Shakhi-Zinda ensemble, which is more than 500 years old.

Shakhi-Zinda is a dual-dome mausoleum (Fig. 1*a*), which, as many scientists believe, lies atop the grave of the astronomer Kazy-zade Rumi, who worked in the Ulugbek observatory. Figure 1*b* displays samples of the ceramic facing of the Shakhi-Zinda ensemble.

The x-ray phase composition of the ceramic facings was studied using the DRON-6 diffractometer (CoK_α radiation) with the sample table rotating at 1 °/min.

To obtain the most complete information on the structure formation in ceramic facings, the microstructure was studied by the “in transmission” method using an ÉMB-100BR electron microscope; the replica is platinum-carbon. Scanning electron microscopy with a Philips 525M SEM was used to investigate the porosity structure of the samples studied.

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Figure 2 displays an x-ray diffraction pattern of the ceramic facings studied.

The characteristic intense lines of quartz (d/n = 0.166, 0.197, 0.228, 0.245, 3.34, and 0.423 nm) can be seen in the diffraction pattern of the powder; lines due to wollastonite (d/n = 0.158, 0.191, 0.208, and 0.305 nm), monticellite (d/n = 0.160 and 0.181 nm), anortite (d/n = 0.177, 0.187, 0.223, 0.252, and 0.375 nm), and feldspar (d/n = 0.285 nm) are present.

As one can see in Fig. 2, new minerals formed in the ceramic facings of the Shakhi-Zinda facings during firing: wollastonite, monticellite, and anortite, which attests to the higher content of calcium oxide in the raw materials used [4].

Electron-microscopic photographs of the samples studied are displayed in Fig. 3.

The electron-microscopic pictures of the samples studied confirm the results of the x-ray phase analysis.

Wollastonite is calcium metasilicate (β-$CaO \cdot SiO_2$) and polymorphic; it crystallizes in two modifications — α and β [5]. The high-temperature α-modification is pseudowollastonite, while wollastonite proper is the low-temperature β-modification. In the opinion of the authors of [6] and according to studies performed by the present author [7], wollastonite creates a complete framework, preventing a change of the previous volume, i.e., it appreciably decreases the stress and shrinkage of ceramic articles.

As one can see in Fig. 3, the wollastonite crystals presented occupy in the samples studied an intermediate position between needle-shaped as described in [5] and elongated

Fig. 1. Part of the Shakhi-Zinda ensemble (*a*) and the ceramic facings studied (*b*) (top two samples in the form of small bricks, and the bottom two in the form of small tiles).

prisms as described in [7]. According to the diagnostics indicators, the wollastonite studied in transmitted light is easily recognized by the refractive indices and the plank-shaped form of the crystals. In reflected light wollastonite can be distinguished from silicate phases of other compositions if and only if its regular crystalline shape is present.

Anortite — a feldspar ($CaO \cdot Al_2O_3 \cdot 2SiO_2$) — is the final member of plagioclases and possesses all properties characteristic of feldspar minerals [7]. This aluminosilicate is polymorphic, and besides anortite two additional unstable modifications are known [7]. It is encountered in nonmetallic minerals only in the stable modification; the melting temperature is 1550°C. The formation of crystalline embryos of anortite and its effect on the increase of strength during firing of ceramic tiles is extremely rarely studied in the literature [1 – 4].

Anortite forms in alumina melts in the presence of calcium oxide and silicon oxide [7]:

$$CaO + Al_2O_3 + SiO_2 \rightarrow CaO \cdot Al_2O_3 \cdot SiO_2.$$

Anortite forms with albite ($NaSi_3AlO_8$) a continuous series of solid solutions, called plagioclases [7].

The authors of [5] observed under a microscope large anortite crystals in the form of small tablets with polysynthetic twins. As Fig. 3 evidences, in the samples studied anortite crystallizes in short-prismatic form; other forms are also encountered on occasion. In transmitted light anortite is easily identified by the index of refraction. In reflected light anortite can be distinguished from quartz and other low-refracting minerals only by its etchability in hydrochloric acid.

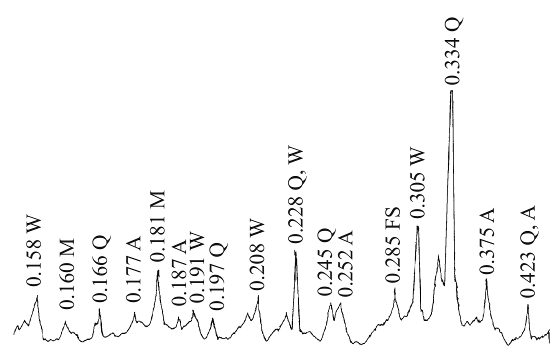

Fig. 2. X-ray diffraction patterns of the facings of the Shakhi-Zinda ensemble: W) wollastonite, M) monticellite, Q) quartz, A) anortite, and FS) feldspar.

Monticellite $CaO \cdot MgO \cdot SiO_2$ belongs to an extensive group of olivines, which are orthosilicates of bivalent metals,

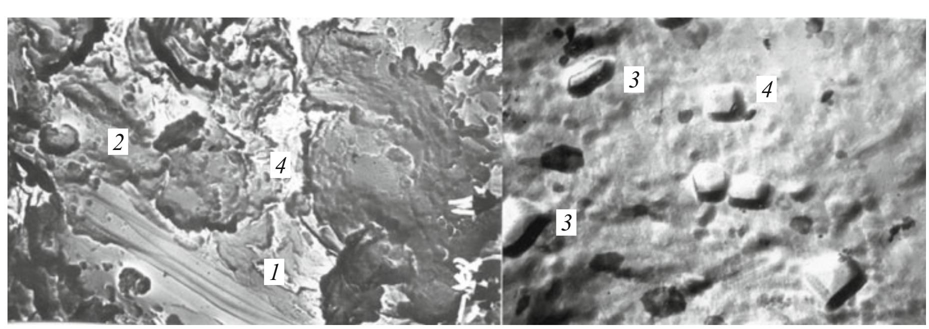

Fig. 3. Electron-microscopic photographs of ceramic facings of the Shakhi-Zinda ensemble: *1*) accumulation of wollastonite crystals with plate-shaped appearance; *2*) single crystals of monticellite of the pseudocubic system; *3*) anortite crystals with short-prismatic habitus; *4*) fused quartz crystals of prismatic and bipyramidal habitus; ×15000.

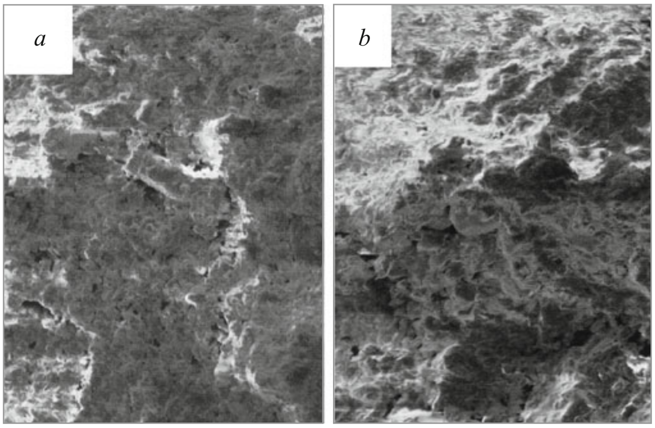

Fig. 4. Character of the porosity of the ceramic facings of the Shakhi-Zinda ensemble: *a*) upper samples, presented in Fig. 1 (bricks); *b*) lower samples, presented in Fig. 1 (small tiles); ×1000.

forming between each other continuous series of solid solutions [5]. It is quite often encountered in nonmetallic inclusions, melts at 1498°C and decomposes at 1300°C [5].

Figure 3 displays monticellite in the pseudocubic system in the ceramic facing ensemble of Shakhi-Zinda. According to the diagnostic indicators monticellite is identified in transmitted light quite easily according to the refractive indices and other constants. In reflected light it is difficult to distinguish from other minerals with close optical properties.

Scanning electron microscopy was used to determine the character of the porosity in the ceramic facings of the Shakhi-Zinda ensemble.

The pores were studied on a fresh cleavage face of the samples under 100-fold magnification (Fig. 4).

As one can see in Fig. 4*a*, the presence of three types of pores is characteristic for the upper samples in Fig. 1: slit-like; isometric, and oddly shaped. In the lower samples (Fig. 1*b*) the character of the porosity is somewhat different, isometric pores predominate, and rounded and oval closed cavities are observed.

The presence of pores and therefore nonuniformities of the material has a deleterious effect on the properties of ceramic articles, the harmful effect on the mechanical strength of elongated (slit-like) pores being estimated as approximately five times greater than for rounded pores [8, 9]. In addition, the presence of slit-like pores implies that the sintering processes did not go to completion [8, 9].

In summary, these studies have shown that wollastonite and anortite are responsible for the strength and durability of the ceramic facings of the Shakhi-Zinda ensemble. In addition, the isometric pores and oval closed pores present in the ceramic facings of the Shakhi-Zinda ensemble impart mechanical strength to the facings.

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